

AD-A248 861



R & D Status Report



(2)

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Program code number: 2G10

Contractor: Center for Materials Research  
Stanford University  
Stanford, CA 94305-4045

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Grant Title : Growth and Evaluation of Nonlinear Optical Materials for  
Laser Applications: Barium Borate, Lithium Borate and Silver  
Gallium Selenide

Key Personnel : Robert S. Feigelson, Principal Investigator  
Roger K. Route, Senior Research Scientist  
Ekkes Brück, Post-doctoral Associate  
N. H. Kim and H. Lee, Graduate Students

Reporting Period : 08-01-90 to 01-31-92

Description of Progress :

BBO and LBO:

A major goal of this program is to develop processes that industry can use to grow large high quality  $\beta$ -barium borate (BBO) and lithium triborate (LBO) crystals with good yield. High optical quality single crystals of both are currently being grown by modified top-seeded solution growth (TSSG) methods using sodium oxide and boric oxide, respectively, as solvents. This advanced TSSG growth technology has been transferred to both INRAD, Inc. and Cleveland Crystals, Inc. during the course of the program.

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The development of a phenomenological model which accurately predicts the growth rate in these systems has allowed us to grow cylindrical boules of both in multi-centimeter lengths, which has not previously been possible by the TSSG method. The crystal growth process is also being monitored by a high resolution weight sensing system which verifies system parameters. Development of a control algorithm using the growth rate model and real time weight sensing is being pursued in order to develop a totally automatic diameter control process.

Additional research has focused on reducing the density of solvent inclusions in flux-grown barium borate by optimizing forced convective stirring in the melts and by understanding the effects of melt chemistry. Computer-controlled periodic reversals in seed rotation have demonstrated that the density of solvent inclusions can be reduced by forced convection, but the underlying causes for solvent incorporation on the growth interface have never been clearly understood. Evidence of carbonate impurities in BBO crystals and solidified charges has recently been discovered, reinforcing previous suspicions of insoluble particulate contamination in the melts, and additional experiments are underway to elucidate the role of carbonate ions that are introduced during the melt synthesis process.

Studies on flux-grown LBO have also focused on improving the yield of useful optical crystals. The mechanism of surface decomposition that results in seed erosion and extensive cracking of the boules upon cooling has been identified and the problem eliminated in the past year. This technology has also been transferred to INRAD, Inc. and a patent disclosure application filed.

We have recently undertaken the metastable growth of barium borate crystals directly from the melt by the Czochralski method. Growth can be carried out at rates 100 times faster than solution techniques permit. These early crystals, the first grown by this method in the U. S., typically consist of several grains and contain minor cracking due to differential stress, but they appear to be free of solvent inclusions. Issues relating to residual strain and control of the seeding process are being investigated.

We have actively supported university graduate research, supplying BBO and LBO crystals for graduate student projects to both Professors R. L. Byer and R. N. Zare. We have also supplied BBO crystals in two different orientations to DARPA for unspecified applications.

Statement A per telecon Dr. Vern Smiley  
NRAD/Code 804  
San Diego, CA 92152-5000

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Accession for	
NRAD	EX-101
BBO TAB	
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#### Infrared NLO Materials:

A second program goal is to identify the causes of laser damage in silver gallium selenide ( $\text{AgGaSe}_2$ ) with the view of improving its power handling capability. We have focused our studies in two areas: improved heat-treatment processes to minimize remnant optical absorption, and the behavior of epitaxially deposited ZnSe as an AR and/or chemical passivation layer. This work is being carried out in close cooperation with STI Optronics, NRL, and R. C. Eckardt in Professor Byer's group.

The heat-treatment processing technology for silver gallium selenide has been extended through a series of chemical interdiffusion studies that has allowed us to determine the kinetics of the heat-treatment process, and to develop a better understanding of the mass transport that occurs. An alternate heat-treatment process was suggested during these studies, and a quantitative comparison is now being carried out.

Using the MOCVD process, we have carried out the first reported heteroepitaxial growth of ZnSe on silver gallium selenide. The initial layers, on the order of 1000 Å thick, were epitaxially oriented with respect to the substrate, but debonding or fracture occurred during cooling. Thinner layers have also shown this behavior, and studies are underway to identify the mechanism(s) responsible. If this problem can be resolved, optical surface absorption measurements and laser damage measurements will be carried out and a comparison made with commercial AR coating performance.

During this reporting interval, zinc germanium phosphide ( $\text{ZnGeP}_2$ ) crystals were grown by the vertical Bridgman method. (These were the first vertically-grown  $\text{ZnGeP}_2$  crystals to be produced in the U. S.) While the vertical Bridgman method allowed greater control over melt stoichiometry than the horizontal process being used in other laboratories, the optical quality of the crystals grown by the two techniques (as indicated by the optical absorption at 1  $\mu\text{m}$ ) was found to be approximately equal. These studies were concluded in favor of a greater emphasis on the borates and silver gallium selenide described above.

#### Fiscal Summary:

- (1.) Amount currently provided on contract : \$ 575,000
- (2.) Expenditures and commitments to date : \$ 329,000